PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM RUBBER SEED SHELL USING ZINC CHLORIDE AS CHEMICAL ACTIVATION

By

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Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

SEPT 2012

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CERTIFICATION OF APPROVAL

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Approved by,

_____

(Mr Azry Borhan)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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SITI HAJAR NAJWA BINTI RAMLI

ABSTRACT

Many human activities generate solid wastes. Large quantities are produced by agriculture and mining, and these wastes generally have big impact on the majority of environment. One of the waste produced by agriculture is rubber seed shell. Rubber seed shell waste consume some spaces in disposal area and a study has been carried out to investigate the application of rubber seed shell as activated carbon that can be use as adsorbent in waste water process.

The solid waste of rubber seed shells can be reuse to produce activated carbon as adsorbent in wastewater treatment. By using Zinc Chloride as activating agent, a few factors of characteristics is studied.

This further study will be done to discover the factor that affecting the adsorption process to be applied in wastewater treatment. Those factors are activation time, activation temperature and size of activated carbon produced. The study is carried out to control the waste produced that gives impact to the environment as well as to reduce the pollution in waste water treatment.

ACKNOWLEDGMENT

First and for most, great gratitude I praise to God for giving me this strength to complete the journey of my student life and to be able for me to undertake this project.

I would also like to give thanks to my supervisor Mr Azry Borhan for his support throughout the project. Without all of his comments and guidance, I would not able to reach up to this point and to finish this project. His time for the evaluation is most appreciated.

Also, my great appreciation to the Head of department, laboratory staffs, technicians and lecturers of Universiti Teknologi Petronas for the helps and updating informations concerning Final Year Project.

Finally, I would like to express my great gratitude to my sponsor PETRONAS, my family, and my friends for giving me continuous supports in both emotionally and financially as well as valuable advices for my project.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Today, rubber seed shells have lots of applications. One of it is to produce activated carbon to be use as adsorbent in wastewater treatment.

An adsorbent is a substance in nature with high -surface area that can absorb onto its surface by intermolecular forces. Only at low concentration in the adsorption isotherm linear, at higher concentration the adsorption isotherm may be Langmuir , Freudlich or BET in nature.

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

Excess waste from rubber production especially rubber seed shells give a big impact on the environment especially disposal problem. There is no effort on commercializing rubber seed shell for technical application.

1.2.2 Significant of the Project

The study is significant in producing activated carbon by using the waste from rubber seed shell. It is one of the most widely used materials due to its low cost. Besides, disposal problem can be solved to an significant product that give beneficial to the environment. The study gives a deep understanding on preparation and characterizing of activated carbon from rubber seed shell.

1.3 OBJECTIVE AND SCOPE OF STUDY

Objective of this project are :

- To reduce the excess waste of rubber seed shells.
- To evaluate the characteristics of activated carbon produced by rubber seed shells.

Scope of study:

This study will discuss on concept of adsorption process including characteristics of adsorbent and factor that affecting the process. Trough out the project, we will discuss about adsorption isotherm and isotherm that will be involve is BET.

1.4 FEASABILITY

It is obligatory for the chemical engineering in final year student to complete the Final Year Project within 2 semesters (FYP1 & FYP2). During first semester, there will be research work regarding the project and followed by experimental work on second semester. For the second semester, the project continuous with the experimental work and test the samples with waste water. The project is feasible within the scope and time frame regardless of no issues with regard to equipment function and material availability.

CHAPTER 2

LITERATURE REVIEW

2.1 RUBBER

According to Project Final Report 2007, the statistic for waste from rubber seed shells produced per year in Malaysia is 19 tonnes per year. Preventing and managing waste is at the heart of sustainable development. Waste implies unnecessary depletion of natural resources, unnecessary costs, and environmental damage. Sustainable waste management is about using resources more efficiently.

Rubber (hevea brasiliensis) tree starts to bear fruits at four years of age. Each fruit contain three or four seeds, which fall to the ground when the fruit ripens and splits. Each tree yields about 800 seeds (1.3 kg) twice a year. Peninsular Malaysia, comprising 12 of the 14 states in the Malaysian federation is among the world's most important rubber growing area. Rubber is also grown in Sabah (formerly North Borneo) and Sarawak, which, known together as East Malaysia. Altogether Malaysia produces almost 20% of the world's natural rubber. More than half Malaysia's rubber comes from thousands of privately owned small landholdings, which are usually about 2 hectares. The rest is grown on big estates owned by various companies; each can cover over a thousand hectares. Altogether, Malaysia has 1.7 million hectares of rubber plantation. A rubber plantation is estimated to be able produce about 800-1200 kg rubber seed per ha per year (Siriwardene and Nugara, 1972), and these are normally regarded as waste. According to the Association of Natural Rubber Producing Countries, Kuala Lumpur, Malaysia has an estimated acreage of 1,229,940 hectares of rubber.

Plantation in 2007 (Malaysian Rubber Board, 2009) Based on an estimated average of 1000 kg seeds per ha/ yr, the projected annual production of rubber seeds in Malaysia would be 1.2 million metric tons.

One of the renewable energy is biodiesel trough transesterification process from rubber seeds. This process combined the extraction and production of biodiesel from the seeds, thus cutting short the process and lowering production cost. The rubber oil is blended with diesel oil as fuel. Rubber seeds were not edible but could be found in abundance in the country, adding that there were 1.2 million ha of rubber plantation in Malaysia. By using oil derived from rubber seeds instead of oil palms could easily avert the debate of a conflict between food and fuel. 1kg of rubber seeds could produce between 300ml and 400ml of biodiesel fuel. It is estimated that in an area of 1ha of rubber trees, there are between 800kg and 1,200kg of rubber seeds. . The estimated availability of rubber seeds in India is about 30,000 tons per annum, which can yield rubber seed oil to the tune of about 5000 tons. (Reksowardojo,Bui)

In Malaysia alone the figure was over 85 percent of the plantations, with a total available timber volume of 543 million m^3, thus indicating a decrease in replanting over time. Unfortunately, this process produced lots of solid waste especially rubber seed shells. This waste could cause significant environment and disposal problems. One of the ways to solve this problem is by reuse the solid waste that is rubber seed shell as activated carbon. Activated carbon is by far the most common adsorbent used in wastewater treatment.(Kishore).

2.2 ADSORPTION

Adsorption is typically used in wastewater treatment to remove toxic or recalcitrant organic pollutants (especially halogenated but also non-halogenated), and to a lesser extent, inorganic contaminants, from the wastewater. Adsorbate or solute is the material being adsorbed (e.g., 2,4,6-trichlorophenol). Adsorbent is the solid material being used as the adsorbing phase (e.g., activated carbon)..

Now day, many industrial wastewaters contain substances that are difficult to remove via conventionally secondary treatment, toxic or hazardous and volatile and cannot be transferred to the atmosphere. The pollutants have the potential for creating noxious vapours or odours, or for imparting colour to the wastewater and present is very small concentrations that make their removal via other methods difficult.

In any adsorption process the material being adsorbed (e.g., a pollutant) is simply but effectively removed from one phase (e.g., a wastewater) and transferred to another phase (e.g., activated carbon). This means that adsorption is a physical separation process in which the adsorbed material is not chemically altered.· Since the chemical characteristics of the adsorbed material are not changed the use of adsorption in wastewater treatment is associated with the removal of hazardous material(s) from the wastewater and its transfer to the activated carbon This implies that the activated carbon now contains the hazardous material. Therefore, appropriate actions must then be taken to treat the spent activated carbon at the end of a cycle. The carbon can be:

- regenerated (i.e., the hazardous material may be removed via stripping)

- disposed of (together with the pollutants it contains) in a landfill

- destroyed (together with the pollutants it contains) in an incinerator

Since, during adsorption, the pollutant is removed by accumulation at the interface between the activated carbon (absorbent) and the wastewater (liquid phase) the adsorbing Capacity of activated carbon is always associated with very high surface area per unit volume.

Pores in activated carbon typically range from 10 to 10,000 Å in diameter.

Classification	Pore diameter* range (nm)	Pore diameter* range (µm)	Pore diameter* range (Å)
Micropores	< 2.0	< 0.002	< 20
Mesopores	2 – 50	0.002 – 0.05	20 – 500
Macropores	> 50	> 0.05	> 500

Table 2. 1:Type of pores

Microspores are primarily responsible for the adsorption characteristics of activated carbon.

The most important factors affecting adsorption are surface area of adsorbent. Larger sizes imply a greater adsorption capacity. Smaller particle sizes reduce internal diffusion and mass transferlimitation to the penetration of the adsorbate inside the adsorbent. However, wastewater drop across columns packed with powdered material is too high for use of this material in packed beds. Addition of powdered adsorbent must be followed by their removal. Contact time or residence time. The longer the time the more complete the adsorption will be. However, the equipment will be larger. Upon contacting an amount of activated carbon with a wastewater containing an absorbable substance adsorption will take place. Adsorption will continue until equilibrium will be established between the substance in solution and the same substance in the adsorbed state. At equilibrium a relationship exists between the concentration of the species in solution and the “concentration” of the same species in the adsorbed state (i.e., the amount of species adsorbed per unit mass of adsorbent).

The adsorption equilibrium relates q to C. The equilibrium is a function of the temperature. Therefore, the adsorption equilibrium relationship at a given temperature is typically referred to as adsorption isotherm, i.e.

q = f (C)

where:

q = mass of species adsorbed/mass of

adsorbent (i.e., equilibrium concentration of

adsorbable species in solid adsorbent)

C = equilibrium concentration of absorbable species in solution different types of equilibrium relationships

It has been found that for most of the cases of importance in wastewater treatment the function q = f(C) takes the form of one of the

following isotherms:

Langmuir isotherm

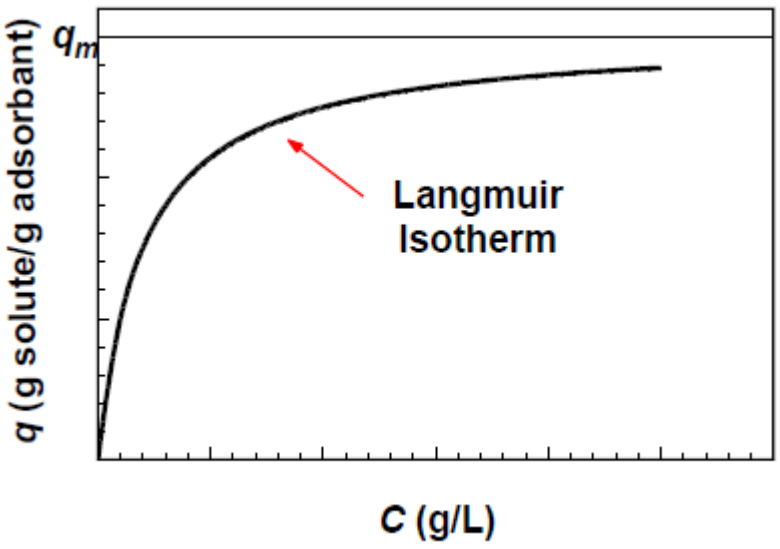

Figure 2. 1:Langmuir isotherm

Brauner-Emmet-Teller (BET) isotherm

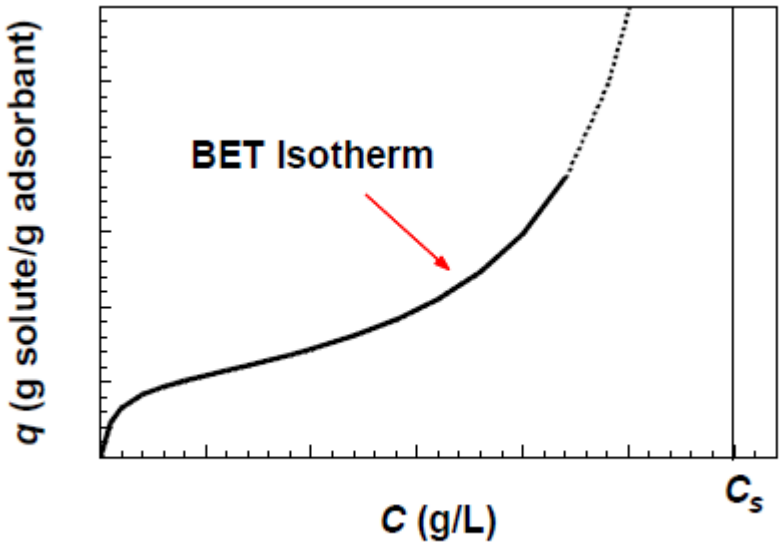

Figure 2. 2:BET Isotherm

Freundlich isotherm

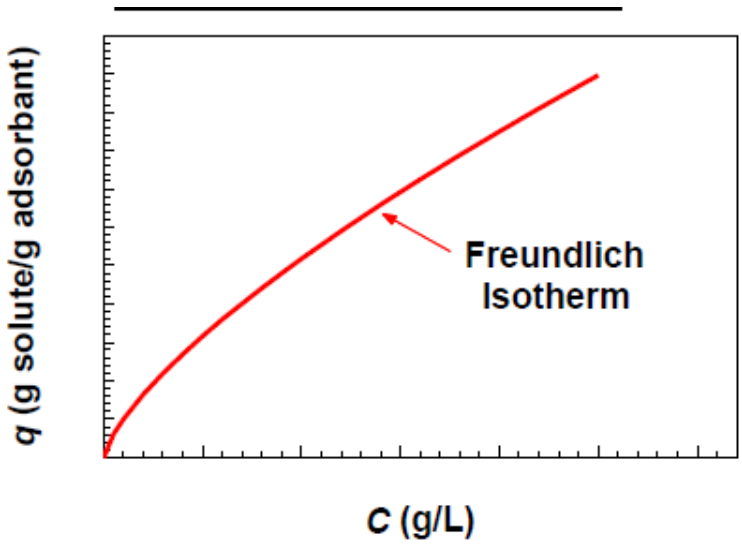

Figure 2. 3:Freundlich Isotherm

2.3 TYPES OF ISOTHERM

2.31 Type i Isotherm

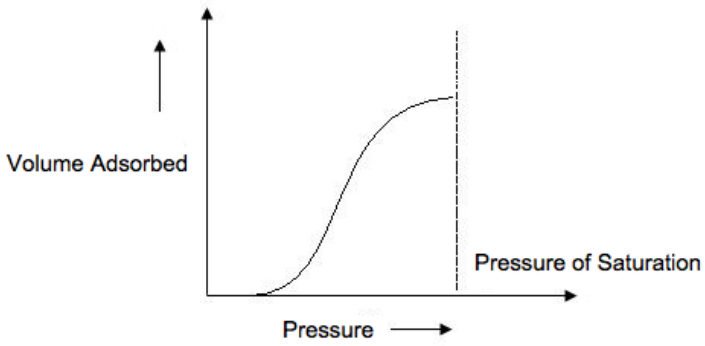

Figure 2. 4:Type I

The above graph depicts Monolayer adsorption.

- This graph can be easily explained using Langmuir Adsorption Isotherm.
- If BET equation, when P/P0<<1 and c>>1, then it leads to monolayer formation and Type I Adsorption Isotherm is obtained.
- Examples of Type-I adsorption are Adsorption of Nitrogen (N2) or Hydrogen (H) on charcoal at temperature near to -1800CTYPE II ISOTHERM

2.32 Type ii Isotherm

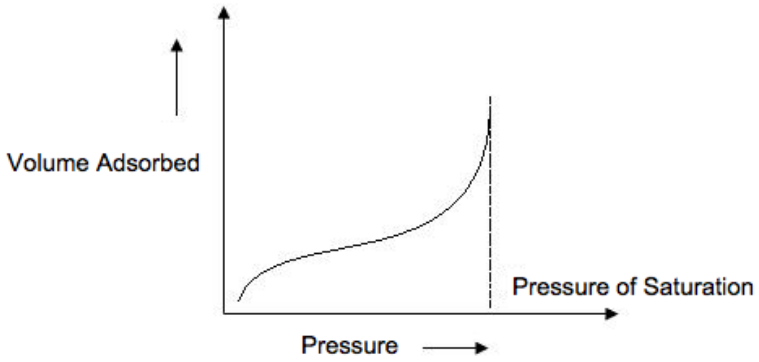

Figure 2. 5:Type II

- Type II Adsorption Isotherm shows large deviation from Langmuir model of adsorption.
- The intermediate flat region in the isotherm corresponds to monolayer formation.
- In BET equation, value of C has to be very large in comparison to 1.
- Examples of Type-II adsorption are Nitrogen (N2 (g)) adsorbed at -1950C on Iron (Fe) catalyst and Nitrogen (N2 (g)) adsorbed at -1950C on silica gel.

2.33 Type iii Isotherm

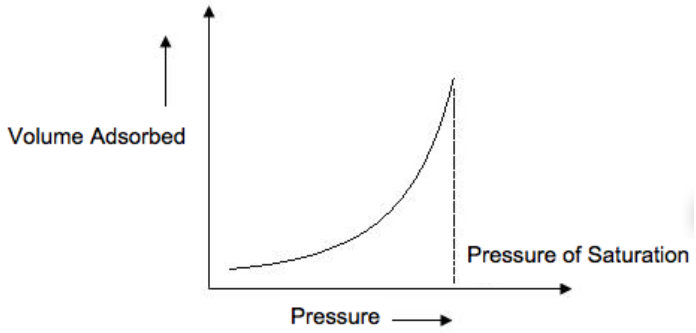

Figure 2. 6:Type III

- Type III Adsorption Isotherm also shows large deviation from Langmuir model.
- In BET equation value if C <<< 1 Type III Adsorption Isotherm obtained.
- This isotherm explains the formation of multilayer.
- There is no flattish portion in the curve which indicates that monolayer formation is missing.
- Examples of Type III Adsorption Isotherm are Bromine (Br2) at 790C on silica gel or Iodine (I2) at 790C on silica gel.

2.34 Type IV Adsorption Isotherm

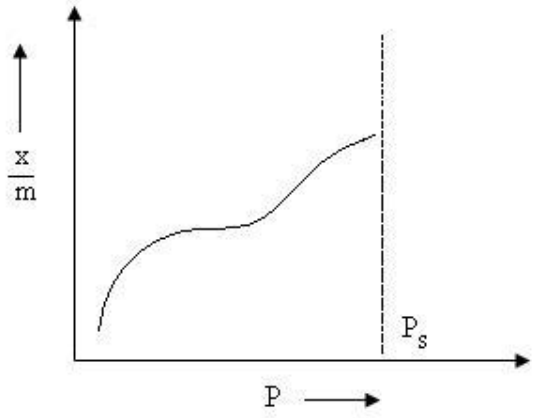

Figure 2. 7:Type IV

- At lower pressure region of graph is quite similar to Type II. This explains formation of monolayer followed by multilayer.
- The saturation level reaches at a pressure below the saturation vapor pressure .This can be explained on the basis of a possibility of gases getting condensed in the tiny capillary pores of adsorbent at pressure below the saturation pressure (PS) of the gas.
- Examples of Type IV Adsorption Isotherm are of adsorption of Benzene on Iron Oxide (Fe2O3) at 500C and adsorption of Benzene on silica gel at 500C.

2.35 Type V Adsorption Isotherm

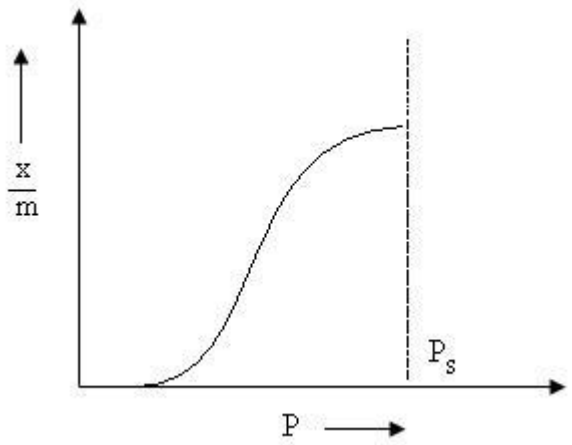

Figure 2. 8 : Type V

- Explanation of Type V graph is similar to Type IV.
- Example of Type V Adsorption Isotherm is adsorption of Water (vapors) at 1000C on charcoal.
- Type IV and V shows phenomenon of capillary condensation of gas.

2.4 USING MTBE AS WASTE TO TEST THE ADSORBENT

Methyl tertiary butyl ether (MTBE) has been used as a fuel additive to increase anti-knock properties in gasoline engines since the 1980s. It replaced very harmful organic lead compounds and aromatic hydrocarbons.

Significant contamination is increasingly being detected in groundwater and surface water used for drinking water due to the many years MTBE has been used and as a result of its particular properties of persistence and high mobility in the environment. MTBE has since been classified as a potential human carcinogen in higher doses and will need to be replaced by environmentally friendly alternatives in the future.

Dealing with the pollution currently created by MTBE, however, remains problematic. Current technologies, including ozonation, microbiological methods, and traditional activated carbon filtration, are not sufficiently able to remove MTBE or the more recent ETBE during waste water or drinking water treatment processing.

The effectiveness of contaminant removal essentially depends on the activated carbon properties. Since the pore size distribution of conventional activated carbon is not variable, the adsorption of many different substances is non-selective. Low-molecular trace elements could previously only be adsorbed in very low concentrations since there was insufficient

CHAPTER 3

METHODOLOGY

3.1 PROJECT FLOW

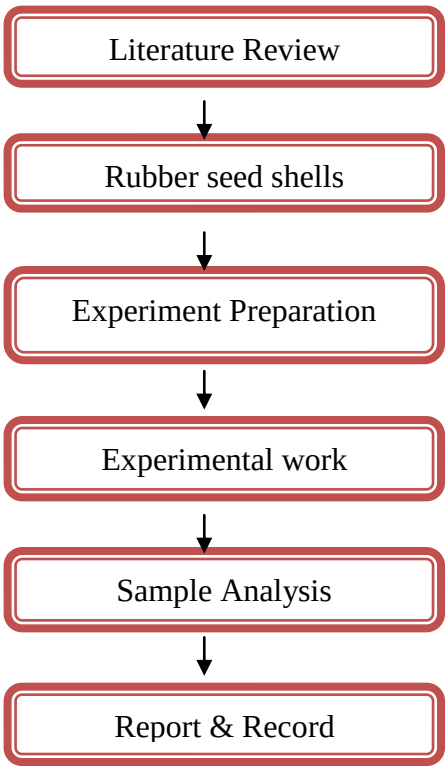

Figure 3. 1: Project Sequence Flow Chart

3.2 EXPERIMENTAL WORK

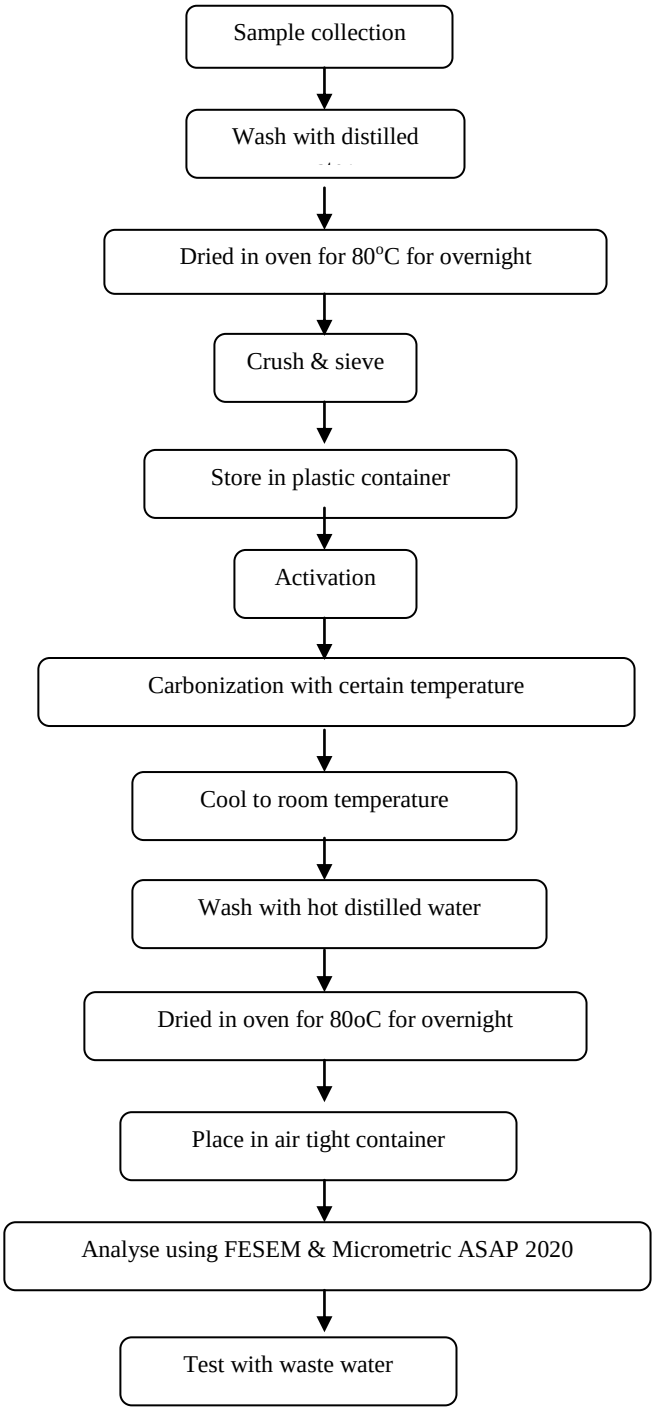

Figure 3. 2: Experimental Work

3.3 PROJECT ACTIVITIES

3.31 Sample Collection

For this project the sample is taken from rubber plantation at Batu Gajah. Figure 3.3 shows the rubber tree at the plantation. Figure 3.4 shows rubber seed shells after being collected and Figure 3.5 shows the raw materials have been cleaned.

Figure 3. 3: Rubber tree

Figure 3. 4: Rubber seed shell shel

Figure 3. 5: Cleaned rubber seed

3.32 Crushed & Sieved

The rubber seed shells have been crushed and sieved according to sizes which are 3mm,500μm & 250 μm.

Figure 3. 6: Siever

Figure 3. 7 : Rubber seed shells crushed&sieved

3.33 Activation

About 10 g of sample was impregnated with $ZnCl_2$.Impregnated can be defined as dry weight of $ZnCl_2$ is allowed to soak with rubber seed shell for overnight for the full adsorption of the reagent to the raw material.13.628g of $ZnCl_2$ is diluted to a 100ml solution for ratio 1:1. For ratio 1:2, 27.256g of $ZnCl_2$ is used.

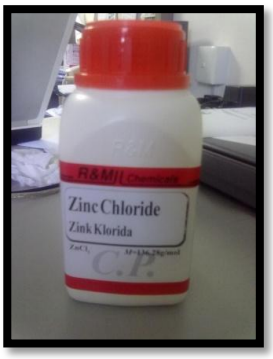

Figure 3. 8 : Zinc Chloride

Figure 3. 9 : Preparation for activation

Figure 3. 10 : Preparation of zinc chloride solution

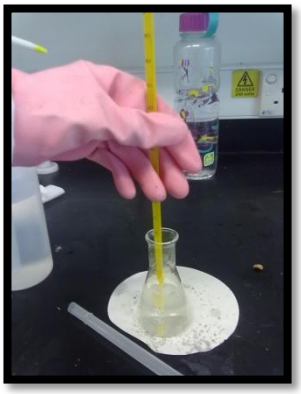

Figure 3. 11 : Activation

3.34 Carbonization

The next process is carbonization by carbonize the impregnated rubber seed shell into fixed bed activation. The temperature is to be set 500^{o}C for 30 minutes under the flow of Nitrogen gas. It is to be estimated for 30 minutes so that the rubber seed shell will turn black and become a dry powder. The activated rubber seed shell is cooled to room temperature and washed with hot distilled water for few times to remove remaining $ZnCl_2$ and dried in oven for 80^{o}C for overnight.

The experiment is repeated by using different size of rubber seed shell(3mm, 500um & 250um),carbonization duration time(30mins, 90mins,and 180mins) and temperature(400^{o}C,500^{o}C & 600^{o}C). The sample is placed in air tight container, sealed with parafilm to be analysed.

Figure 3. 12 : Furnace for carbonization

Figure 3. 13 : Preparation to carbonized

Figure 3. 14 : After being carbonized

Figure 3. 15 : Sample to be send for analysis

3.4 ACTIVITIES/GANTT CHART AND MILESTONE

No	Detail/Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	■	■	■	■	■	■	■									
2	Submission of Progress Report									●							
3	Project Work Continues									■	■	■	■	■			
4	Pre-EDX												●				
6	Submission of Draft Report													●			
4	Submission of Dissertation (soft bound)														●		
8	Submission of Technical Paper														●		
9	Oral Presentation															●	
9	Submission of Project Dissertation (Hard Bound)																●

Mid-Semester Break

Table 3. 1 : Gant Chart

3.5 TOOLS REQUIRED

3.51 Field-Emission SEM (FESEM)

Nanometre-resolution imaging in field-emission SEM (FESEM) instruments is now widely used in materials characterization. The use of a high-brightness field-emission gun makes it possible to acquire nanometre-resolution surface images at low voltages (<5kV). The advantages of low-voltage FESEM include enhanced surface sensitivity, reduced sample charging for no conducting materials, reduced damage of delicate samples, and significantly reduced electron range and interaction volume in bulk samples ("High-resolution and low-voltage," 2000).

3.52 Micrometric ASAP 2020

Accurate and precise surface area and porosimetry measurements are essential to the determination of the effectiveness and quality of a wide variety of materials. The Micromeritics ASAP 2020 integrates multiple gas sorption techniques into a single, convenient table top instrument.

The ASAP 2020 provides maximum versatility over a remarkable range of applications. Sophisticated system features include:

- Two independent vacuum systems allowing simultaneous preparation of two samples and analysis of another
- A two-station intelligent degas system or fully automated degassing with precisely controlled heating profiles
- Intuitive and powerful Windows-based software that includes easy-to-use interactive Wizards™ to help guide you through even the most challenging experiments

- A highly flexible and interactive reporting system that includes an extremely versatile graphic user interface allowing custom presentation of results.

The pore size distribution, specific surface area and porosity of the sample were determined by nitrogen adsorption-desorption isotherms characterised with Micrometric ASAP 2020 apparatus, using N_2 as the adsorbate. Before the analysis, the samples were degassed under N_2 flow at 350^oC for 2 hours in a vacuum of 27^oC. The specific surface of area was determined by using BET method. The Barret-Joyner-Halenda (BJS) model was used to calculate the pore size distribution. (Rouquerol et al.,1999).

CHAPTER 4

RESULT & DISCUSSION

Size Ratio	3mm	500um	250um	Duration time(min)	Temperature (^{o}C)
1:1	A1	B1	C1	30	400
	A2	B2	C2		500
	A3	B3	C3		600
	A4	B4	C4	90	400
	A5	B5	C5		500
	A6	B6	C6		600
	A7	B7	C7	180	400
	A8	B8	C8		500
	A9	B9	C9		600
1:2	A10	B10	C10	30	400
	A11	B11	C11		500
	A12	B12	C12		600
	A13	B13	C13	90	400
	A14	B14	C14		500
	A15	B15	C15		600
	A16	B16	C16	180	400
	A17	B17	C17		500
	A18	B18	C18		600

Table 4. 1 : List of samples

4.1 FESEM ANALYSIS

Activated carbon is a porosity enclosed by carbon atoms. For this project, the activation carbon is made from rubber seed shell. Activated carbon is a unique material because of the way it is filled with 'holes' the size of molecules. Although they are spaces of zero electron density, these pores posses intense van Der Waals forces and these are responsible for the adsorption process. Activated carbon must possess a large volume of micropores, with an appropriate pore size distribution in order to adsorb molecules of different sizes.

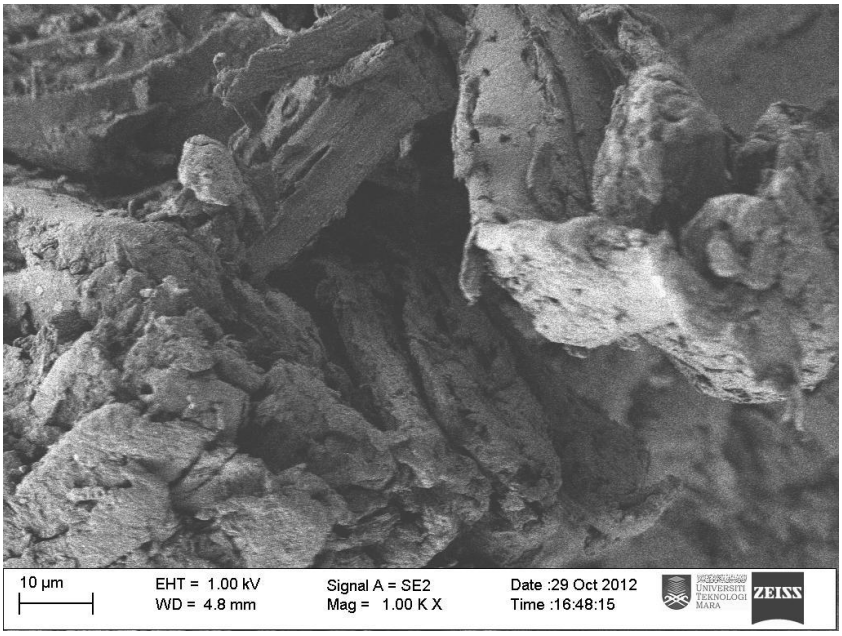

Figure 4. 1 : Raw material of 500um

This is raw material of 500um. The structure showed it has a good texture for preparing activation carbon because it potential to create a pore for the adsorption. Size of a particular cell is dependent on the surface area/volume relationship. A bigger cell possesses a relatively smaller surface area, and vice versa.

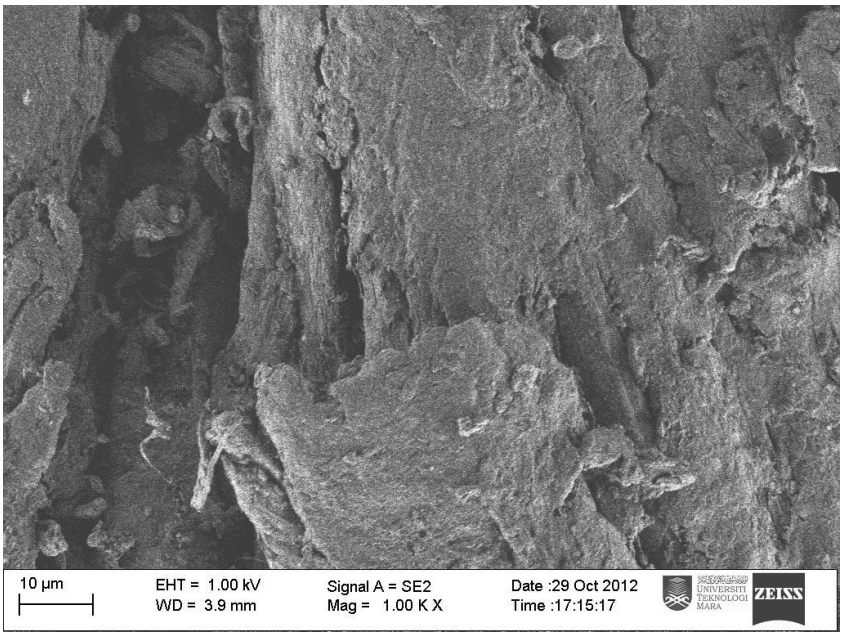

Figure 4. 2 : Raw material of 250um

This is raw material for 250um. It shows that the structure of raw material is smaller and surface area is larger. This will give a bigger effect of absorption than 500um. Any molecule which enters into this space is subject to intense dispersion forces from the carbon atom which make up the surface of the space. These forces can trap the molecule for periods of time much longer than on an open surface and so the phenomenon of physical adsorption is generated. The site where a molecule can be adsorbed is called adsorption site. The smaller size of molecules can enhance the adsorption process.

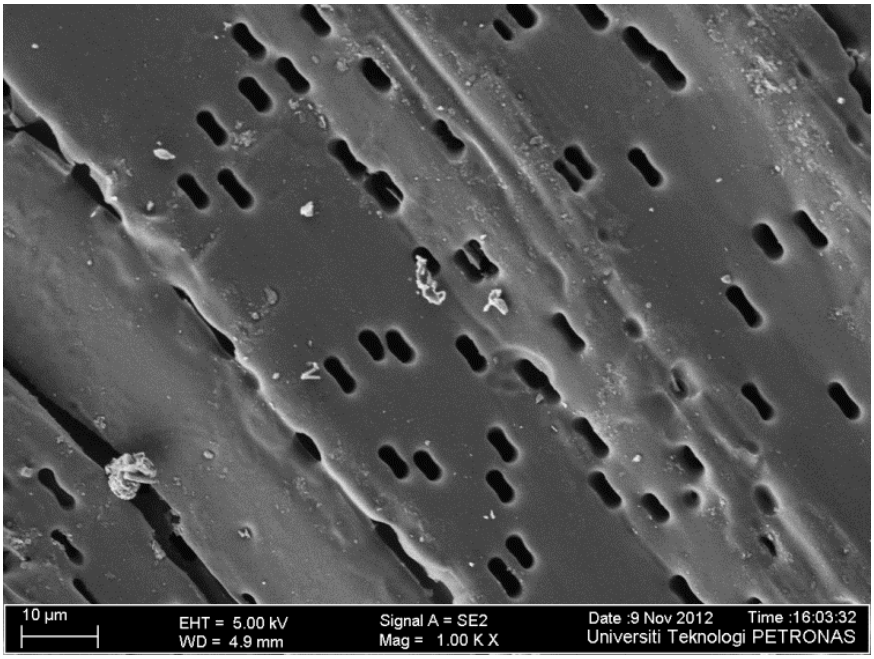

Figure 4. 3 : Sample C2

This is carbonized sample with ratio 1:1 at 500^{o}C for 30 minutes. The pores are seems to expend and create a canal for the adsorption. This is the early stage as many clearly pores are found. The effect of the carbonization sample is a slightly weight loss. Impregnation may lead to fragmentation of cellulose and other component in sample. Dehydration is possible because the chemical is a liquid at the temperature of the process thus facilitating the bonding to the sample being thermally degraded. The sample is able to transfer the water to the reactant in the reacting mass to form hydrated compound which then loses water with increasing temperature.

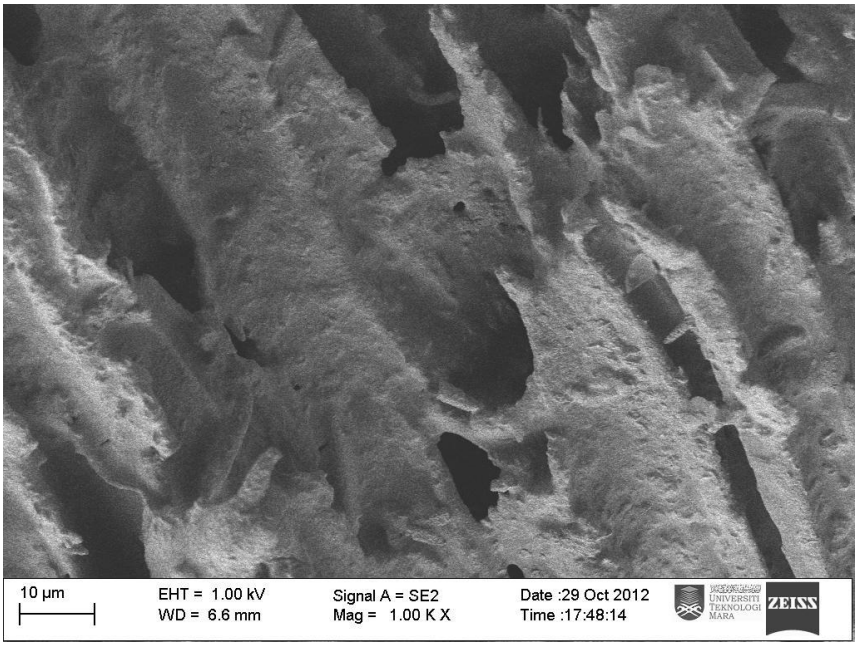

Figure 4. 4 : Sample C11

This is sample of C11 at 500^{o} C after 30 minutes with ratio of 1:2. As the ratio of $ZnCl_2$ was increased, so the pore volume increased. As compared to the sample C2, the volume of the pores is smaller than sample of C11. This is the proof that the volume of the pores is increase with the increase of ratio. Thus, this will lead to the increase efficiency of the adsorption.

At this early stage, there is evidence of chemical and physical change. This reaction is further accompanied by dehydration, degradation and condensation. The primary reaction leads to a reduction in weight.

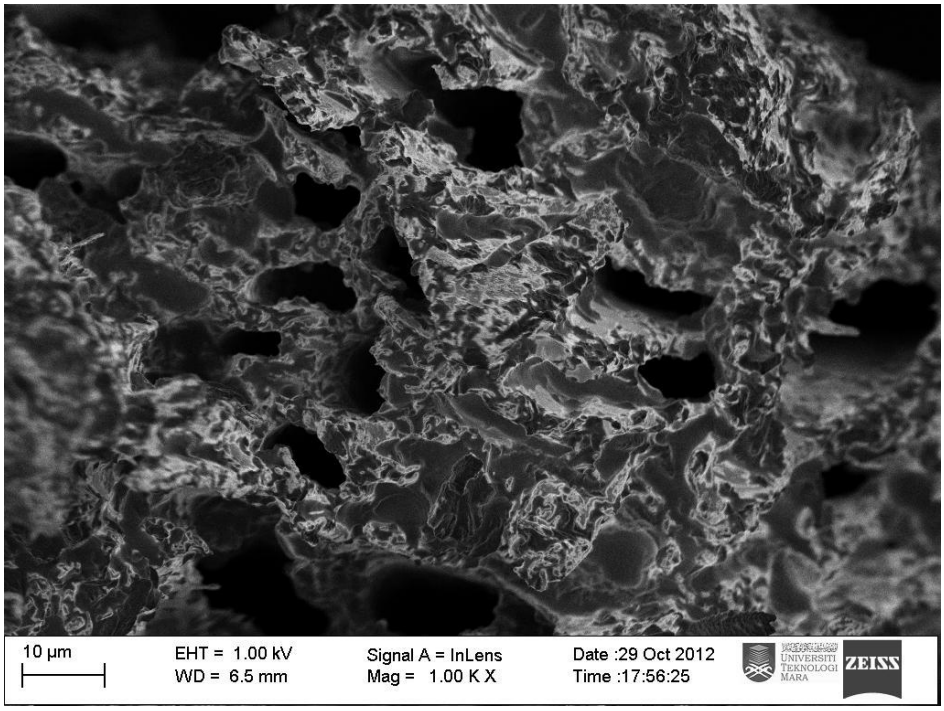

Figure 4. 5 : Sample C14

This is sample of C14 at 500^{o} C after 90 minutes with ratio of 1:2. As time is longer, the pores are well developed and lead to large surface area and porous structure of the activated carbon. The activated carbon is matured enough to absorb particles.

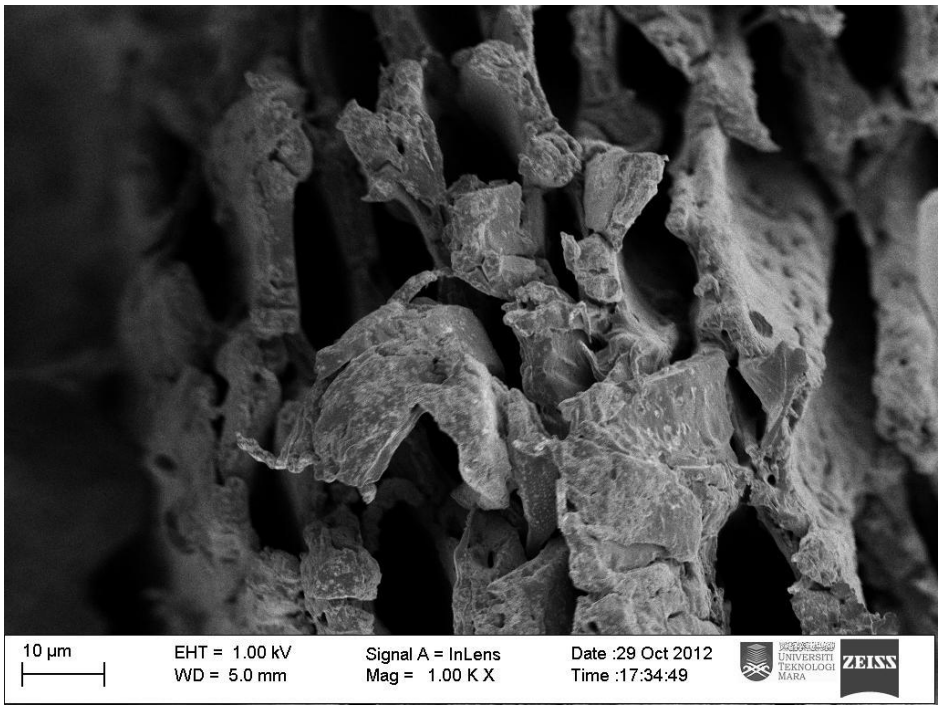

Figure4. 6 : Sample of C18

This is the sample of C18 at 600^{o}C at 180 minutes with ratio 1:2. The pores are found to be expended and partially broken due to excessive duration of high temperature exposure. The volume of the pores is decrease due to the contraction of the wall.

4.2 BET ANALYSIS

From BET analysis, the result obtained were specific surface area, total pore volume and average pore diameter. Tables below show that the value of the collected data according to the size of sample. The result from Figure 4.6 shows that the distribution of the size for sample 500um with different time of carbonization, ratio, and temperature.

4.21 Bet Analysis For Size 250um

Table 4. 2:Result for 250um

sample	Time (min)	temperature (^{o}C)	Ratio	Specific surface area,$S_{BET(m}{}^{2}g^{-1})$	Total pore volume,$V_T(cm^3g^{-1})$	Average pore diameter,D(um)
C2	30	500	1:1	1023.43	0.63	1.83
C11	30	500	1:2	1027.37	0.67	1.90
C14	90	500	1:2	1096.99	0.72	2.32

From the result, we can see that the highest value of specific surface area is 1096.99m^2g^{-1} with total pore volume of 0.72cm^3g^{-1} and with pore diameter of 2.32um. This is the result from sample C14 which has been carbonized for 500^oC for 90 minutes and the ratio of the chemical activated agent to the raw rubber seed shell is 2 :1, means that the amount of Zinc Chloride as chemical activated agent is double than the rubber seed shell. From this result, this adsorbent is mesopore as the pore diameter is more than 2um. According to the researches, the optimum temperature for carbonization is between 400oC-500oC.

For sample C2 and C11,both of them undergoes same duration for carbonization and temperature which are 500^oC and 30 minutes but the only manipulated variable is ratio of chemical activated agent(zinc chloride). For sample C2,the ratio is 1:1 whereas for sample C11,the ratio is 1:2 As we the result obtained, the value of specific surface area is 1023.43m^2g^{-1} with total pore volume of 0.63cm^3g^{-1} and with pore diameter of 1.83um for sample C2. For sample C11, the value of specific surface area is 1027.37m^2g^{-1} with total pore volume of 0.67cm^3g^{-1} and with pore diameter of 1.90um. As we can see, the distribution size for sample C11 is more than C2 as the ratio of the sample C11 is double than C2. This is the proof that the volume of the pores is increase with the increase of ratio. Thus, this will lead to the increase efficiency of the adsorption.

Both of samples C2 and C11 are micropore adsorbent because their pore diameter is less than 2.0um

4.22 Bet Analysis For Size 500um

Table 4. 3:Result for 500um

sample	Time (min)	temperature (^{o}C)	Ratio	Specific surface area,$S_{BET(}m^{2}g^{-1}$)	Total pore volume,$V_{T}(cm^{3}g^{-1})$	Average pore diameter,D(um)
B1	30	400	1:1	1020.91	0.61	1.91
B10	30	400	1:2	1021.56	0.62	1.95
B14	90	500	1:2	1093.71	0.68	2.28
B18	180	600	1:2	1058.76	0.64	2.22

The table above shows that the value for specific surface area, total pore volume and average pore diameter for the sample size of 500um. The temperature, ratio and the duration time for carbonization is varies.

From the table, the highest value for specific surface area is 1093.71 $m^{2}g^{-1}$ with total pore volume of 0.68 $cm^{3}g^{-1}$ and average pore diameter of 2.28um. This sample has undergone the process of 500^{o}C of carbonization for 90 minutes with the ratio of 1:2.

At this point, the adsorbent is well developed to mesopore and the cell of it is matured for the adsorption. This adsorbent is mesopore as the pore diameter is 2.28um which is more than 2.0um. This temperature, ratio and duration of carbonization is optimum for this adsorbent.

The comparison between sample B1 and B10 is reasonable because both of them had undergo the same temperature of carbonization which is 400^{o}C and the duration is 30 minutes. I could compare the ratio of both sample which is sample B1 has a ratio of 1:1 and the sample of B10 has a ratio of 1:2. The ratio is chemical activated agent to the rubber seed shell. This comparison shows that the increment in ratio of chemical activated agent will increase the pore diameter as well as surface area and total volume.

Sample B18 has carbonization temperature of 600^{o}C with duration of 180 minutes. The value obtained is 1058.76 m^2g^{-1} of specific surface area , with total pore volume of 0.64 cm^3g^{-1} and average pore diameter of 2.22um. The size of the sample is getting smaller due to the excessive temperature and duration for carbonization. The sample size is reduced because the cell is destroyed by the high excessive temperature for a long time. This shows that, this temperature and duration is not optimum for the adsorbent.

By comparing both of the table, which are the size of the sample are different, we can conclude that the smaller size of the adsorbent will give bigger surface area, pore volume and pore diameter for the more efficient adsorption. Besides, the optimum temperature for the carbonization is 500^{o}C with duration time of 90 minutes. The ratio is one of the factors that will affect the adsorption efficiency.

4.23. Relative Pressure Vs Volume Adsorbed

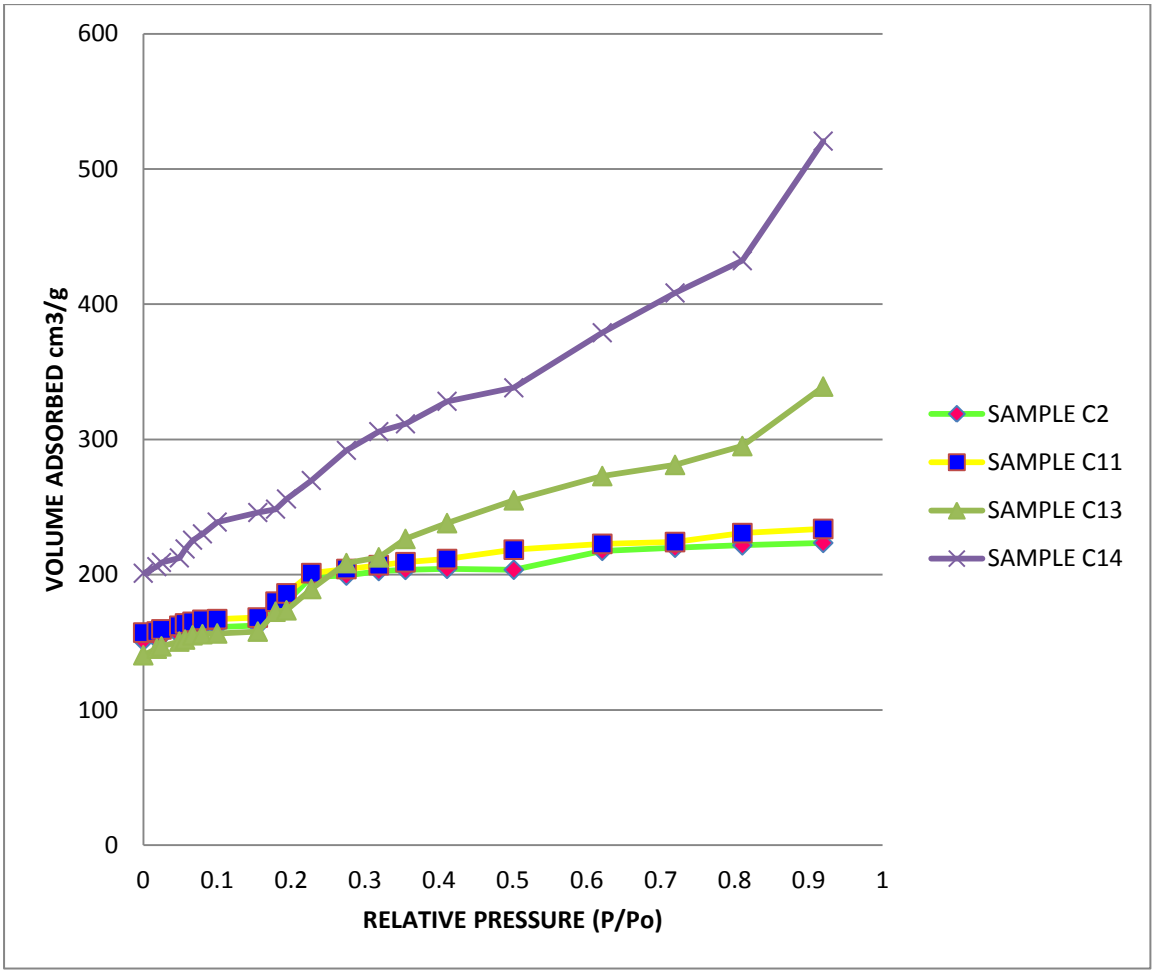

Figure 4. 6:Relative pressure vs Volume adsorbed

The figure above shows that the adsorption-desorption isotherms of the selected 4 samples obtained from different time and temperature. The quantity of the adsorbed Nitrogen is plotted vs relative pressure.

For sample C2 and C11, these samples are prepared for 30 minutes of carbonization with 500^{o}C of temperature and the ratio is 1:1 and 1:2. Based on the IUPAC classification of isotherm, this corresponds to Type I of the BET isotherm. According to Harry Marsh in Activated Carbon, the adsorbent from Type I reach a maximum value of adsorption without inflections and are characteristic of activated carbon has microporosity only. The gradients of the initial part of the isotherm from p/p^{o} values from 0-0.05, are indicative of the dimensions the microporosity. The steeper the gradient, the narrower the micropores.

For sample C13, this activated carbon is prepared for 400^{O}C for 90 minutes with ratio of 1:2. Based on the graph, the sample is in the group of Type II of BET isotherm. This type of isotherm shows an inflection in the region of $p/p^{o} > 0.1$, and the high relative pressure, $p/p^{o} > 0.9$, where the extents of adsorption on open surfaces with multilayer formation occurring in the final stage of the process. This type of adsorbent has mixed characteristic of micropores and open surface.

For sample C14 that undergoes a carbonization of 90 minutes with the temperature of 500^{o}C correspond to the type III of BET isotherm base on the graph plotted. This type of isotherm is convex, looking upwards and mesopore has been developed.

4.24 Pore Diameter Vs Pore Volume Graph

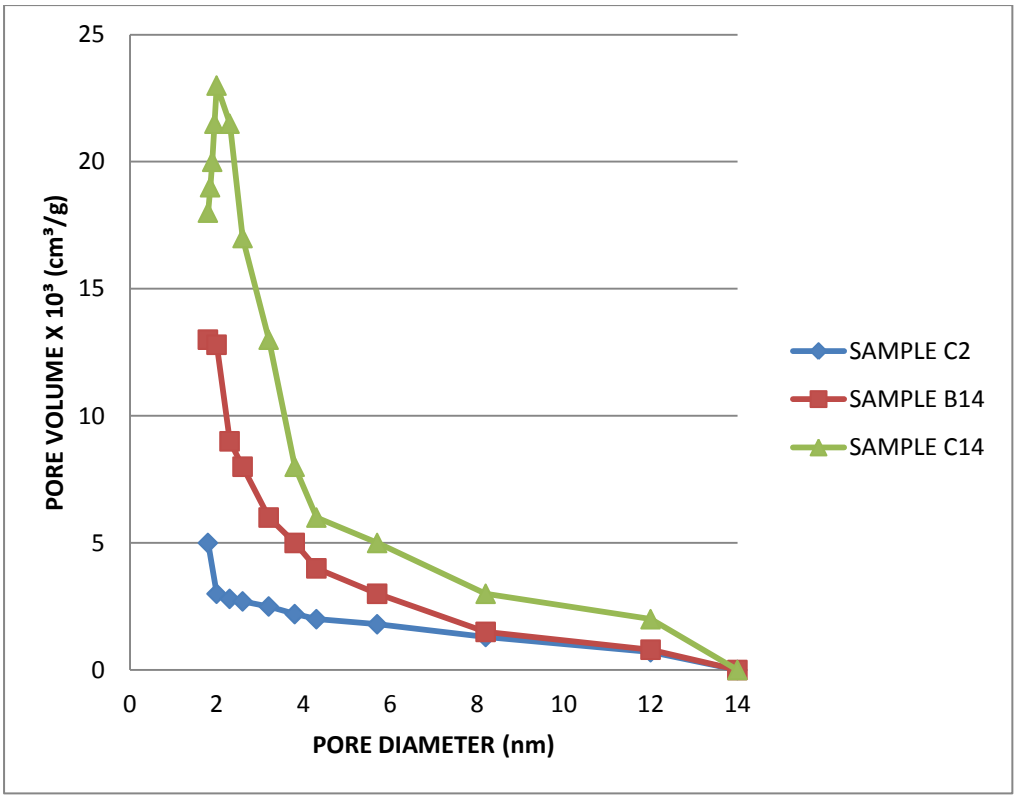

Figure 4. 7:Pore diameter vs Pore volume

From the graph above, the pore volume of sample C2, B14 and C14 is plotted against pore diameter.

For sample C2, the pore diameter is 1.83Á that indicates the adsorbent is micropore. At this stage, the pores are in the early development.

For sample B14 and C14, the pore diameter are 2.28Á and 2.32Á which are mesopores.

4.3 REMOVAL EFFICIENCY

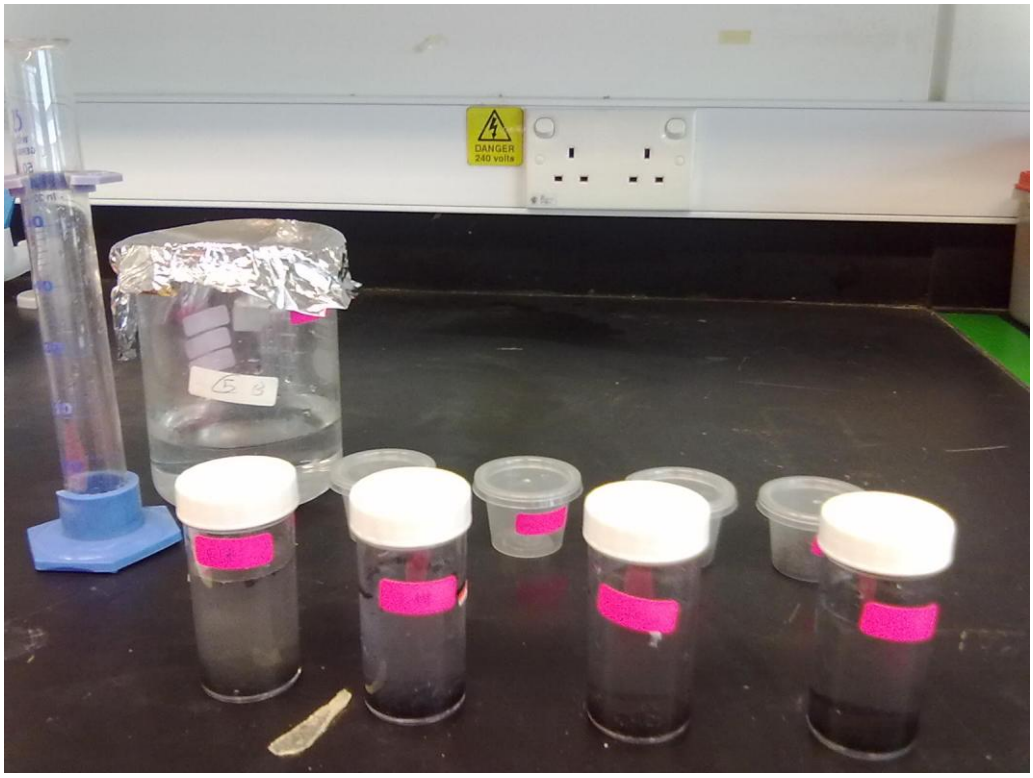

Figure 4. 8:wastewater of MTBE

The adsorbents were tested with the wastewater that containing MTBE of the concentration of 0.4mg/L. This test was conducted for 5 days and the data were taken every 2 days. The table below shows that the removal efficiency of the adsorbent from MTBE with different temperature, duration of carbonization and ratio.

Standard : 0.4mg/L

Table 4. 4: Removal Efficiency percentage

DAYS	1		3		5	
	Concentration (mg/L)	% of removal	Concentration (mg/L)	% of removal	Concentration (mg/L)	% of removal
C2	0.3272	18	0.301	24.75	0.283	29.25
C11	0.2863	28	0.26	35	0.24	40
C14	0.2045	48.9	0.179	55.25	0.15	62.5
C18	0.398	0.5	0.387	3.25	0.38	5

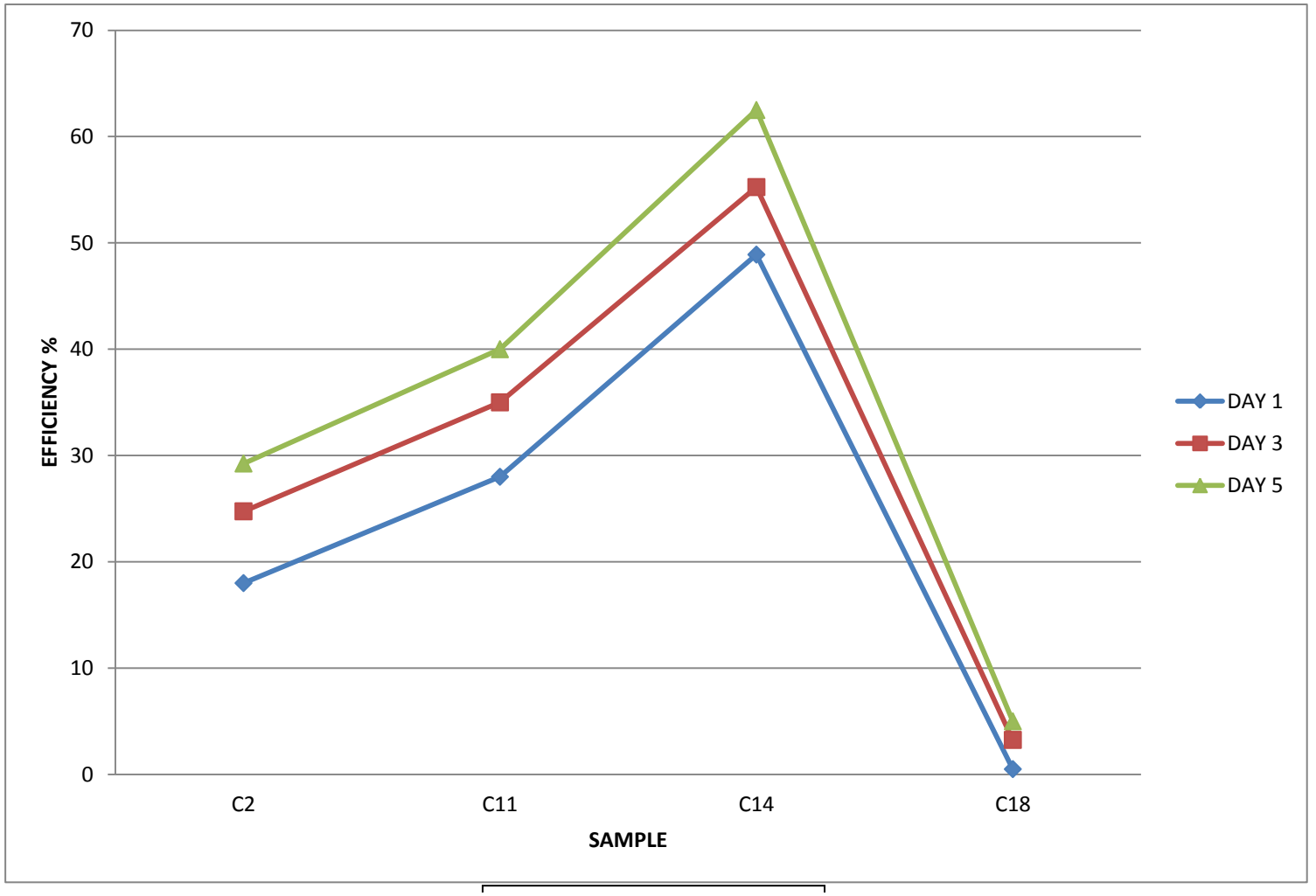

Figure 4. 9:Removal Efficiency

The wastewater containing 0.4mg/L was tested with the sample C2, C11, C14 and C18. At first, 5g of adsorbent was submerged in the labelled wastewater. For the interval of 2 days, the concentration of wastewater is tested and the percentage of the removal efficiency is calculated by using this equation :

$$\text{Percentage of removal efficiency} = \frac{\text{initial conc} - \text{final conc}}{\text{Initial conc}} \times 100\%$$

The result shows that, the percentage of removal efficiency of sample C14 is the highest for the 5 days. The sample C14 is mesopore and the temperature, duration and the ratio is optimum for this adsorbent. This means, this adsorbent gives the best result among all for the highest efficiency of adsorption.

The result for C2 and C11 once again shows that the ratio of chemical actived agent which is zinc chloride is affect the removal efficiency. The sample of C11 has a higher percentage of removal than the sample C2.

The sample C18 has the least removal efficiency percentage which is 5% only after 5 days. This indicated that the excessive of carbonization temperature and duration destroyed the cell in the adsorbent as well as the efficiency is the least.

CHAPTER 5

CONCLUSION & RECOMENDATION

The study is relevant with the objective which is to evaluate the characteristics of activated carbon produced by rubber seed shells. The method used in this project is chemical activation through zinc chloride as activating agent. The rubber seed shell is able to to produce an adsorbent.

The characteristics of the rubber seed shell as adsorbent by using zinc chloride as activated agent has been found. The project proves that the optimum temperature for the carbonization is 500^0C with duration time of 90 minutes. The smaller size of the adsorbent gives the higher efficiency for the adsorption of wastewater. Rubber seed shell is able to absorb MTBE as waste water. The increment ratio of the chemical activated agent increase the efficiency of adsorption as well as the pore diameter is bigger. The mesopore and micropore development are suitable as the activated carbon for adsorption process.

The objectives of this project have been successfully achieved by determining the characteristics of the rubber seed shell as adsorbent by using zinc chloride as chemical activated agent.

RECOMMENDATION

It is recommended that the study is continue by using other source as wastewater and to varies the ratio with the chemical activated agent. The study is suggested to be done on the effect of the impurities in the waste water.

Besides, the laboratory of BET and FESEM should be done earlier so that the student will able to proceed with more samples. The chosen sample for the test need to be appropriate with the expected result.

Environmental care and safety should be first priority for the project and individual. Safety precautions and rules that has been regulated must be followed. This project is one of the solutions for the pollution problem that related to wastewater.

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APPENDIX

Result from BET

C12		C11		C13		C14	
P/PO	VOLUME	P/PO	VOLUME	P/PO	VOLUME	P/PO	VOLUME
0	150.99	0	157.17	0	140.449	0	201.04
0.018283638	152.4531	0.01828364	157.9543	0.018284	145.302	0.018284	205.915
0.024555645	154.5278	0.02455564	159.5278	0.024556	147.024	0.024556	209.043
0.04879644	155.453	0.04879644	162.194	0.048796	150.54	0.048796	212.4039
0.056555578	158.4512	0.05655558	163.9217	0.056556	152.042	0.056556	219.0186
0.066688717	160.0673	0.06668872	165.1062	0.066689	155.2053	0.066689	225.4915
0.0799908	161.1265	0.0799908	166.611	0.079991	155.983	0.079991	230.1903
0.09999912	161.3254	0.09999912	167.104	0.099999	156.543	0.099999	238.9136
0.154884758	162.3314	0.15488476	168.2811	0.154885	157.9332	0.154885	245.9611
0.178955778	175.1744	0.17895578	180.0164	0.178956	172.612	0.178956	248.4517
0.193733367	179.9234	0.19373337	186.1295	0.193733	173.673	0.193733	255.9891
0.22734567	198.48	0.22734567	201.1294	0.227346	189.403	0.227346	269.639
0.27479436	199.356	0.27479436	204.183	0.274794	208.563	0.274794	291.942
0.3185567	202.712	0.3185567	207.018	0.318557	212.831	0.318557	305.776
0.354678	203.54	0.354678	209.194	0.354678	226.4622	0.354678	311.551
0.41096455	204.34	0.41096455	211.5491	0.410965	238.123	0.410965	328.194
0.50125653	203.65	0.50125653	218.4912	0.501257	255.014	0.501257	338.19
0.6209512	217.62	0.6209512	222.7693	0.620951	272.941	0.620951	378.916
0.7194035	219.954	0.7194035	224.1055	0.719404	281.1823	0.719404	408.331
0.81044822	221.71	0.81044822	230.712	0.810448	295.1054	0.810448	432.1932
0.919800453	223.5	0.91980045	233.8143	0.9198	339.1045	0.9198	520.612